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NOTIFICATION OF ELECTION

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(54) Title: MICROWAVE CURABLE COMPOSITIONS

(57) Abstract: The present invention discloses a curable composition that is controllably curable by microwave energy. More particularly, this invention discloses a heat curable composition that is controllably curable by microwave energy including at least one heat curable resin component, microwave absorbable particles in an amount of about 10 % by weight of said composition and at least one curing agent for the heat curable resin component. A method of controllably curing a composition that includes the inventive composition is also contemplated.

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MICROWAVE CURABLE COMPOSITIONS

FIELD OF THE INVENTION

The present invention relates to microwave curable compositions and to methods of
5 controllably heating and curing heat curable compositions.

BRIEF DESCRIPTION OF RELATED TECHNOLOGY

Conventional techniques of heat curing compositions include the use of hot air, hot press
and radiant heat. The equipment currently needed to perform these curing methods has safety
10 and cost concerns. Such equipment also uses a great deal of energy in operation, in the form of
heat which does not assist in curing the material. Such equipment is usually bulky, taking up
costly manufacturing space and requires a great deal of maintenance and safety attention.
These heating curing methods can be time consuming, especially when thick sections are cured
and it takes a long time for the heat to transfer throughout the polymeric composition. Also,
15 these methods may cure sections of a thick article unevenly, leaving sections uncured and
degrading other sections.

Microwave energy is a heating means that advantageously increases the speed and
lowers the power consumption of heating an article. However, use of microwave energy has
20 been limited as a heating means for polymeric compositions because much of the microwave
energy passes through the polymeric composition, i.e., the microwave energy is poorly
absorbed by the polymeric compositions. Thus, heat from microwave energy will not be
evenly distributed throughout the article and parts of the article heated with microwave energy
may begin to degrade before the other portions of the article are heated.

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Efforts to use microwave energy to heat compositions have had limited success.
Attempts to use microwave energy to distribute heat evenly to compositions include U.S.
Patents 5,188,256 to Nottingham, et al. and U.S. Patent No. 5,368,799 issued to Haas et al.,
which are incorporated herein by reference. These patents describe a hot melt apparatus where

microwave absorbable particles are incorporated into a composition that absorbs microwave energy and converts to heat which melts a neighboring thermoplastic composition.

Additionally, U.S. Patent No. 5,446,270 discloses the use of ferromagnetic particles in amounts up to 10% of the total composition, that have been dispersed or positioned in a matrix to be heated by microwave energy in general microwave applications. U.S. Patent 5,317,045 issued to Clark, Jr. et. al., discloses the application of microwave energy to remotely heat a polymeric material to a selected temperature. Specifically, Clark describes the use of thermosetting polymers to form joints in composite structures like those found in graphite composite frames of state of the art aircraft. Although these patents describe the use of ferromagnetic properties to absorb microwave energy and subsequently heat a polymeric material, they have had limited use in curing compositions due to uneven distribution of particles and the difficulty in uniformly curing a composition. The Clark patent is limited in its disclosure and teachings to low amounts of particles, particles from iron-lignosulfonate and to certain types of polymers and applications.

It would be desirable to provide compositions which can uniformly and controllably heat and cure with microwave energy. Additionally, it would be desirable to provide a heating method that cures heat curable compositions faster and more uniformly than conventional heating techniques, reducing the cost and improving the safety of the curing process.

SUMMARY OF THE INVENTION

In one aspect of the present invention there is provided a composition which is controllably heatable and curable by a microwave energy. The compositions include at least one heat curable resin component; microwave absorbable particles present in an amount of about 10% by weight of the composition, and at least one curing agent for the heat curable resin component. The microwave absorbable particles are selected such that the Curie temperature of the particles is higher than the curing temperature of the heat curable resin component. Application of microwave energy to the composition results in the microwave particles absorbing the energy, and converting it to radiant heat energy which rapidly cures the heat curable resin component.

In another aspect of the present invention there is a method for controllably curing a composition. This method includes the steps of selecting appropriate microwave absorbable

particles having a desired Curie temperature; combining the microwave-absorbable particles with a heat curable resin component, which has a curing temperature lower than the Curie temperature and is substantially transparent to microwave energy and the particles are present in an amount of about 10% by weight of the composition; and applying a sufficient amount and wavelength of microwave energy to the heat curable resin component to achieve the desired heating temperature.

The use of microwave absorbable particles to cure the compositions permits formation of articles such as gaskets or seals in minutes rather than hours. Additionally, this method provides uniform and controllable curing throughout the thickness of the composition, achieving requisite seal strengths without a hot press. By heating only the composition with the microwavable absorbent particles, and not the surrounding environment, i.e., air and equipment, results in energy conservation and improved safety. In fact, microwave energy source may be located at a remote distance from a seal to be cured, which greatly simplifies the curing process.

DETAILED DESCRIPTION OF THE INVENTION

The curable resin component used in the present invention may be a functionalized monomer, polymer, and/or oligomer which, in the presence of a curing agent, cures. Examples of heat curable resins include acrylics, elastomeric synthetic rubbers, epoxies, polyesters, polyurethanes, polybutadienes, cyanate esters, bismoleimides, polyimides, phenolics, alkyls, amino resins, silicones, acrylic silicones, epoxy silicones and copolymers and combinations thereof. Other useful curable resin components further include elastomeric deformable materials such as urethane, silicone-acrylate, urethane-acrylate, co-polymers and combinations thereof. One particularly useful heat curable resin is silicone elastomers, such as those used to construct gaskets for automobiles. The curable composition may be in a liquid, film or putty-like form which cures when exposed to heat.

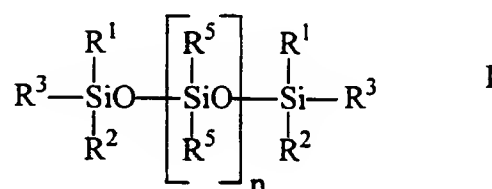
The heat curable resin component may be used in amounts of about 20% to about 95% by weight, and desirably about 40% to about 60% by weight of the total composition.

Useful curable resin components include, but are not limited to, linear or branched polyorganosiloxanes, polydimethylsiloxane chains having crosslinking sites located at either

the ends or the middle of chains, cyclic siloxanes and combinations thereof. Radicals from thermal decomposition of peroxides crosslink polyorganosiloxanes with or without unsaturation. Saturated polyorganosiloxanes may result in a slower curing process than unsaturated polyorganosiloxanes. Particularly useful silicones include vinyl-terminated polysiloxanes. More particularly, dimethyl vinyl silyl terminated polydimethylsiloxanes (10,000 MW vinyl fluid and 62,000 MW vinyl fluid) have been found to be useful in the present invention as a heat curable resin component.

Polyorganosiloxanes having olefinic unsaturation should contain at least one reactive functional group, and desirably two reactive functional groups. More than two reactive functional groups are also advantageous in achieving certain cured properties. The number and type of functional group or groups present can be varied according to the desired properties of the final silicone composition. The organic groups of the polyorganosiloxane are monovalent hydrocarbon radicals and preferably the organo groups include alkyl radicals, such as methyl, ethyl, propyl, etc.; alkenyl radicals such as vinyl, allyl, etc.; cycloalkyl radicals such as cyclohexyl, cycloheptyl, and; mononuclear aryl radicals such as phenyl, ethylphenyl; and haloalkyl such as 3, 3, 3-trifluoropropyl.

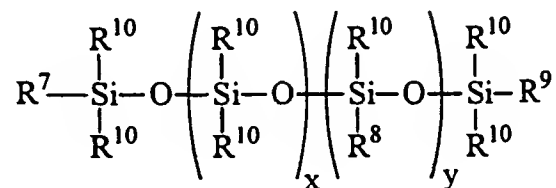
The polyorganosiloxanes of the present invention have the general formula:



wherein R^1 , R^2 , R^3 and R^4 may be the same or different and are substituted or unsubstituted hydrocarbon or hydrocarbonoxy radicals from C_{1-20} , provided that at least one of these R groups, and desirably more than one, include unsaturated functional groups which participate in heat curing, such as vinyl, (meth)acrylate, carboxylate, maleate, cinnamate and combinations thereof. For example, when one or more of the aforementioned R groups (R^1 , R^2 , R^3 and R^4) is not one of the required reactive functional groups, they can be chosen from alkyl radicals such as methyl, ethyl, propyl, butyl and pentyl; alkenyl radicals such as vinyl and allyl; cycloalkyl radicals such as cyclohexyl and cycloheptyl; aryl radicals such as phenyl, methylphenyl, ethylphenyl; arylalkyl radicals such as beta-phenylethyl; alkylaryl radicals; and hydrocarbonoxy radicals such as alkoxy, aryloxy, alkaryloxy, aryalkoxy, and desirable methoxy, ethoxy or hydroxy, and the like. Any of the foregoing radicals may have some or all of the hydrogen atoms replaced, for example, by a halogen such as fluorine or chlorine. One or more of the aforementioned R groups can also be hydrogen, provided the required reactive functional group is present as indicated and the presence of the hydrogen does not deleteriously interfere with the ability of the polyorganosiloxane to perform in the present invention. R^3 in the above formula desirably is a vinyl group or a dimethyl vinyl group.

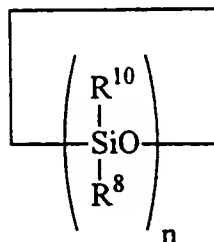
The number of repeating units in the polyorganosiloxanes can be varied to achieve specific molecular weights, viscosities and other chemical or physical properties. Additional reactive polyorganosiloxanes are disclosed in U.S. Patent No. 5,212,211 to Welch II et al., which is incorporated by reference herein, may be used in the present invention. Generally n is an integer such that the viscosity is from about 25 cps to about 2,500,000 cps at 25°C, such as when n is from 1 to 1,200.

The silicon hydride crosslinker may be selected as the curing agent and conforms to the formula below:



wherein at least two of R^7 , R^8 and R^9 are H; otherwise R^7 , R^8 and R^9 can be the same or different and can be a substituted or unsubstituted hydrocarbon radical from C_{1-20} such hydrocarbon radicals including those as previously defined for formula I above; thus the SiH group may be terminal, pendent or both; R^{10} can be H and can also be a substituted or unsubstituted hydrocarbon radical from C_{1-20} such hydrocarbon radicals including those as previously defined for formula I above, and desirably is an alkyl group such as methyl; x is an integer from 10 to 1,000; and y is an integer from 1 to 20. Desirably, R groups which are not H are methyl. The silicon hydride crosslinker should be present in amounts sufficient to achieve the desired amount of crosslinking and desirably in amounts of about 1 to about 10% by weight of the composition.

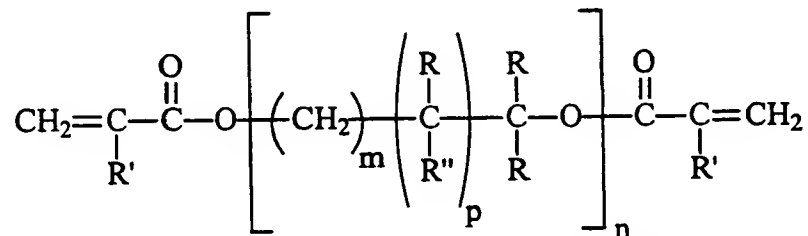
Other silicon hydride crosslinkers are also contemplated including those of the formula:



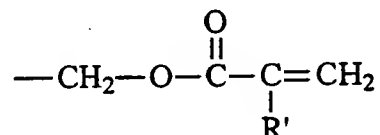
wherein R^8 and R^{10} can be the same or different and can be H or a substituted or unsubstituted hydrocarbon radical from C_{1-20} such hydrocarbon radicals including those as previously defined for formula I above and n is an integer from 3 to 30. R^{10} desirably is an alkyl group such as methyl. Desirably, R groups which are not H are methyl.

Examples of useful (meth)acrylic compositions include, without limitations those described in U.S. Patent Nos. 3,043,820, 3,425,988, 4,103,081 and 4,262,106, all of which are

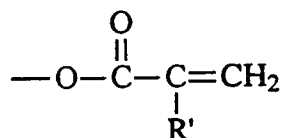
herein incorporated by reference. An example of (meth)acrylic compositions useful in the present invention are those which correspond to the following general structure:



- 5 wherein R represents a radical selected from the group consisting of hydrogen, lower alkyl of 1-4 carbon atoms, hydroxy alkyl of 1-4 carbon atoms, and



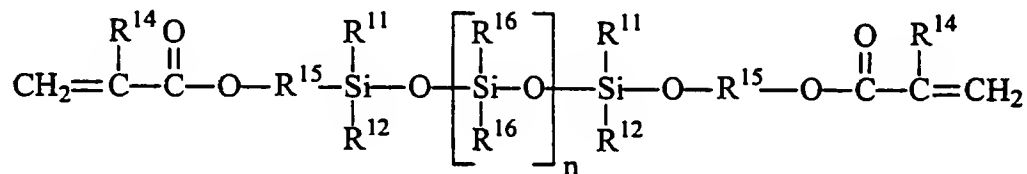
- 10 wherein R' is a radical selected from the group consisting of hydrogen, halogen, and lower alkyl of 1-4 carbon atoms; R'' is a radical selected from the group consisting of hydrogen, -OH and



- 15 wherein m is an integer equal to at least 1, e.g., from 1 to 8 or higher, and preferably from 1 to about 4; n is an integer equal to at least 1, for example, 1 to 20 or more; and p is 0 or 1.

Useful acrylic functional silicone compositions include for example, those disclosed in Loctite U.S. Patent Numbers 4,503,208, 4,528,081, 4,699,802 and 4,675,346, the disclosures of which are incorporated herein by reference. Among the acrylic silicone compositions disclosed therein are those which include compounds having the chemical formula:

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wherein n is an integer from about 50 to about 2000, R¹¹ may be H, alkyl, amino, oxime or -OOCR¹³; R¹² is R¹¹ or hydrocarbyl such as alkyl, aryl, substituted aryl, alkoxy, aryloxy or acrylic groups; R¹³ is hydrocarbyl, R¹⁴ is H or methyl, R¹⁵ is an organo group having a carbon atom bound to the adjacent silicon atom and R¹⁶ is a hydrocarbyl group, such as substituted alkyl, aryl or substituted aryl groups. Specific examples of R¹¹ and R¹² groups are alkyls, such as methyl and ethyl, halo groups such as trifluoropropyl, phenyl and benzyl. R¹¹ and R¹² may also include moisture curable groups such as methoxy. Desirably, R¹³ and R¹⁴ are methyl. More specifically, R¹⁵ may be alkylene, alkyleneoxy, alkenylene of 3-10 carbon atoms, such as propylene and propenylene or arylene groups. Such siloxane compounds may have molecular weights which vary, but desirably are at least about 1,000.

When silicone compositions are employed in the heat curable resin component of the present invention, these compositions may employ hydrosilation addition chemistry to cure. Hydrosilation addition cure is a catalyzed heat cure reaction which uses the presence of one or more unsaturated groups in the heat curable silicone resin component. Examples of heat curable silicone resin components include, but are not limited to polyorganosiloxane containing ethylenically unsaturated functionalities and monomeric silanes containing ethylenically unsaturated functionalities. For example, vinyl terminated polydimethylsiloxane, tetramethyl tetravinyl cyclotetrasilane or tetrakis(vinyl dimethylsilyl)silane may be employed.

When heat curable silicone resins are employed in the present invention, a hydrosilation cross-linker are employed along with a hydrosilation catalyst. Hydrosilation cross-linkers include, but are not limited to, linear polyorganosiloxanes, monomeric silanes,

polydimethylsiloxane chains having SiH functionality located at either the ends or the middle of the chains and cyclic siloxanes. Desirably, the first silicon containing material is selected from the group consisting of SiH terminated polydimethylsiloxane, copolymers of polydimethyl siloxane and polymonomethyl siloxane, tetrakisdimethylsilylsilane, and tetramethyl cyclotetrasiloxane.

The polymerizable polyacrylate esters utilized in accordance with the invention and corresponding to the above general formula are exemplified by, but not restricted to, the following materials: di-, tri- and tetraethyleneglycol dimethacrylate; dipropyleneglycol dimethacrylate; polyethyleneglycol dimethacrylate; di(-pentamethyleneglycol) dimethacrylate; tetraethyleneglycol diacrylate; tetraethyleneglycol di(chloroacrylate); diglycerol diacrylate; diglycerol tetramethacrylate; tetramethylene dimethacrylate; ethylene dimethacrylate; neopentylglycol diacrylate; and trimethylolpropane triacrylate.

While di- and other polyacrylate esters have been found particularly desirable, monofunctional acrylate esters (esters containing one acrylate group) also may be used, such as those having a relatively polar alcoholic moiety. Such materials are less volatile than low molecular weight alkyl esters and, more important, the polar group tends to provide intermolecular attraction in the cured polymer, thus producing a more durable sealant or adhesive. Desirably, the polar group is selected from labile hydrogen, heterocyclic ring, hydroxy, amino, cyano, and halogen polar groups, more specific examples of which are cyclohexylmethacrylate, tetrahydrofurfuryl methacrylate, hydroxyethyl acrylate, hydroxypropyl methacrylate, t-butylaminoethyl methacrylate, cyanoethylacrylate, and chloroethyl methacrylate.

Curable polyolefinic monomers may be used as the heat curable resin component, including acrylic and methacrylic resins, vinyl monomers, unsaturated polyesters solubilized in vinyl monomers and mixtures thereof. Particularly useful curable resin components are ethoxylated trimethylolpropane triacrylate, trimethylol propane trimethacrylate, dipentaerythritol monohydroxypentacrylate, pentaerythritol triacrylate, ethoxylated trimethylolpropane triacrylate, 1,6-hexanedioldiacrylate, neopentyl glycoldiacrylate, pentaerythritol tetraacrylate, 1,2-butylene glycoldiacrylate, trimethylolpropane ethoxylate tri(meth)acrylate, glyceryl propoxylate tri(meth)acrylate, trimethylolpropane tri(meth)acrylate,

dipentaerythritol monohydroxy penta(meth)acrylate, tri(propylene glycol) di(meth)acrylate, neopentylglycol propoxylate di(meth)acrylate, 1,4-butanediol di(meth)acrylate, polyethyleneglycol di(meth)acrylate, triethyleneglycol di(meth)acrylate, butylene glycol di(meth)acrylate, ethoxylated bisphenol A di(meth)acrylate and combinations thereof.

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Epoxy resins useful in the compositions of the present invention include polyepoxides curable by elevated temperature. Examples of these polyepoxides include polyglycidyl and poly(β -methylglycidyl) ethers obtainable by reaction of a compound containing at least two free alcoholic hydroxyl and/or phenolic hydroxyl groups per molecule with the appropriate epichlorohydrin under alkaline conditions or, alternatively, in the presence of an acidic catalyst and subsequent treatment with alkali. These ethers may be made from acyclic alcohols such as ethylene glycol, diethylene glycol, and higher poly(oxyethylene) glycols, propane-1,2-diol and poly(oxypropylene) glycols, propane-1,3-diol, butane-1,4-diol, poly(oxytetramethylene) glycols, pentane-1,5-diol, hexane-2,4,6-triol, glycerol, 1,1,1-trimethylolpropane, pentaerythritol, sorbitol, and poly(epichlorohydrin); from cycloaliphatic alcohols such as resorcinol, quinitol, bis(4-hydroxycyclohexyl)methane, 2,2-bis(4-hydroxycyclohexyl)propane, and 1,1-bis(hydroxymethyl)-cyclohex-3-ene; and from alcohols having aromatic nuclei, such as N,N-bis(2-hydroxyethyl)aniline and p,p'-bis(2-hydroxyethylamino)diphenylmethane. Or they may be made from mononuclear phenols, such as resorcinol and hydroquinone, and from polynuclear phenols, such as bis(4-hydroxyphenyl)methane, 4,4'-dihydroxydiphenyl, bis(4-hydroxyphenyl) sulphone, 1,1,2,2-tetrakis(4-hydroxyphenyl)ethane, 2,2-bis(4-hydroxyphenyl)propane (otherwise known as bisphenol A), 2,2-bis(3,5-dibromo-4-hydroxyphenyl)propane, and novolaks formed from aldehydes such as formaldehyde, acetaldehyde, chloral, and furfuraldehyde, with phenols such as phenol itself, and phenols substituted in the ring by chlorine atoms or by alkyl groups each containing up to nine carbon atoms, such as 4-chlorophenol, 2-methylphenol, and 4-t-butylphenol.

Poly(N-glycidyl) compounds include, for example, those obtained by dehydrochlorination of the reaction products of epichlorohydrin with amines containing at least two amino-hydrogen atoms, such as aniline, n-butylamine, bis(4-aminophenyl)methane, and bis(4-methylaminophenyl)methane; triglycidyl isocyanurate; and N,N'-diglycidyl derivatives of cyclic alkylene ureas, such as ethyleneurea and 1,3-propyleneureas, and of hydantoins such as 5,5-dimethylhydantoin.

Epoxide resins having the 1,2-epoxide groups attached to different kinds of hetero atoms may be employed, e.g., the N,N,O-triglycidyl derivative of 4-aminophenol, the glycidyl ether-glycidyl ester of salicylic acid, N-glycidyl-N'-(2-glycidyloxypropyl)-5,5-
5 dimethylhydantoin, and 2-glycidyloxy-1,3-bis(5,5-dimethyl-1-glycidylhydantoin-3-yl)propane.

Such epoxies are available from a variety of commercial sources, such as the EPON series from Shell Chemical Co., the EPI-REZ series from Rhône-Poulenc, the Araldite series
10 from Ciba-Geigy, the D.E.R. series from Dow Chemical Co., and the EPOTUF series from Reichhold.

Also useful are halogenated epoxy resins such as the brominated epoxides available from the sources shown above. Halogenated epoxy resins in combination with other fire
15 retardant materials may be suitable for use as fire retardant additives in the compositions of the present invention.

Especially preferred epoxy resins useful in the present invention are the diglycidyl ethers of bisphenol A marketed under the tradenames EPON 825 and EPON 828 available
20 from Shell Chemical Co., D.E.R. 331 and 332 available from Dow Chemical Co., and the cycloaliphatic epoxy resin marketed as ERL-4221 by Union Carbide Co.

Various epoxies such as the glycidyl ethers marketed as the EPODIL series by Pacific Anchor Chemical Corporation, a division of Air Products and Chemicals Inc., may be added as
25 epoxy diluents, to reduce the viscosities of the resins of the present invention.

The type of curing agent is chosen based on the choice of heat curable resin component. A curing agent is generally employed to initiate or otherwise promote the curing of the polymer or monomer. Useful curing agents include an amine-containing compound, an azo
30 compound and a peroxide curing agent. In the case of (meth)acrylic compositions, peroxy initiators are generally employed. In the case of heat curing silicone compositions, platinum compositions are often employed.

Curing agents commonly employed in the present compositions are often included in an amount less than about 20% by weight of the composition. Desirably, they are employed in lower levels such as 0.025% to about 10%, desirably about 1 to about 5% by weight of the total composition.

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Peroxy initiators useful in (meth)acrylic compositions include the hydroperoxy curing agents and most preferably the organic curing agents having the formula ROOH, wherein R generally is the hydrocarbon radical containing up to about 18 carbons, desirably an alkyl, aryl or aralkyl radical containing up to about 12 carbon atoms. When warmed to a specific temperature, peroxide curing agents decompose exothermically into free radicals with a half-life period which is characteristic for each peroxide. In the presence of polymerizable double bonds, these free radicals add themselves on thereto and initiate the polymerization.

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Typical examples of such hydroperoxides include cumene hydroperoxide, methylethylketone hydroperoxide, para-menthane hydroperoxide, tertiary butyl hydroperoxide as well as hydroperoxides formed by the oxygenation of various other hydrocarbons such as methylbutene, cetane and cyclohexane. Other peroxy initiators such as hydrogen peroxide or materials such as organic peroxides or peresters which hydrolyze or decompose to form hydroperoxides such as tertiary butyl perbenzoate may also be employed. Organic peroxides, for example percarbonates, diacyl peroxides, per-esters, per-acids or ketone hydroperoxides, are used to a great extent as initiators for free-radical polymerization.

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Other known initiators of free radical polymerization such as saccharin and a variety of secondary and tertiary organic amines are useful in the inventive compositions. A common combination of these initiators involves a hydroperoxide, saccharin and aromatic amine system. The use of metal catalysts, alone or in combination with other conventional accelerators are also useful.

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Various conventional heat-activated curing agents for epoxies are useful in the present invention including imidazoles, preferably 2-ethyl-4-methyl imidazole, 1-(2-cyanomethyl)-2-ethyl- α -4-methylimidazole and 2-phenyl-4,5-dihydroxymethyl imidazole; aliphatic cycloaliphatic amines, preferably 2,2'-dimethyl-4,4'-methylene-bis(cyclohexylamine) (Ancamine 2049); aromatic amines, preferably 4,4'-diaminodiphenyl sulfone (Ancamine S and

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Ancamine SP); a blend of aromatic and aliphatic amines (Ancamine 2038); Lewis Acid catalysts such as boron trifluoride:amine complexes, preferably BF_3 :benzyl amine (Anchor 1907), BF_3 :monoethyl amine (Anchor 1948) and liquid BF_3 :amine complex (Anchor 1222); Lewis Base catalysts such as t-amines, preferably tris(dimethyl-aminomethyl)phenol (Ancamine K.54), dimethylaminomethyl phenol (Ancamine 1110); dicyandiamides, preferably dicyandiamide (Amicure CG). The Ancamine, Anchor, and Amicure series are tradenames for heat activated curing agents marketed by Pacific Anchor Performance Chemicals Division of Air Products and Chemicals, Inc.

Especially pertinent to the anhydride resin compositions of the present invention are the acid anhydride epoxy curing agents. These include, preferably, methyltetrahydrophthalic anhydride, methylhexahydrophthalic anhydride, chlorendic anhydride, and nadic methyl anhydride and mixtures thereof. Nadic methyl anhydride is available as AC-methyl from Anhydrides and Chemicals, Inc.

It will be noted that anhydride based catalysis of epoxy polymerization is an inherently slow process. Accordingly, the resin compositions of the present invention generally employ a minor amount of amine accelerators of the anhydride catalysis, preferably benzyl dimethylamine; 2-ethyl-4-methyl imidazole, available as Imicure EMI-24 from Pacific Anchor; and 2,4-diamino-6[2'-methylimidazolyl-(1)]ethyl-s-triazine isocyanurate adduct.

Accelerators may be used to affect the cured properties of the compositions in areas of adhesion and sensitivity to oxygen and to speed curing of the composition. Amines and organic sulfimides are examples of useful accelerators. Among the useful amines include amine oxides, sulfonamides and triazines. The use of accelerators may be in amounts of about 0.1 to about 5, preferably about 1 to about 2, percent by weight of the total composition.

Catalysts may be added to the curable composition to help promote curing. A platinum catalyst is useful for addition curing systems. The platinum catalyst may be solid platinum, deposited on a carrier such as charcoal or gammaalumina. Any type of platinum catalyst compatible with the present invention may be used. The catalyst may be utilized in the amounts from about 0.02% to about 20% of the total composition.

It is generally desirable that the catalyst is a solubilized platinum catalyst complex.

Useful catalyst and curing agent solutions include methyl hydrogen polysiloxane solution with a 25% platinum catalyst solution, cyclo tri(vinyl methylsiloxane) with a $\text{Pt}(\text{CO})_2\text{Cl}_2$ solution and combinations thereof in amounts of 0.02-20.0% and desirably 0.02-5.0% by weight of the solution.

Other classes of catalysts include, in addition to organoplatinum and organoplatinum complexes, organorhodium and platinum alcoholates. Complexes of ruthenium palladium, osmium and iridium are also contemplated. Organoplatinum catalysts are particularly useful herein. Of the non-platinum based catalysts useful, those based on rhodium are most preferred. The organometallic hydrosilation catalysts may be used in any effective amount to effectuate thermal curing. Combinations of various precious metal or precious metal-containing catalysts are contemplated. The amount of this catalyst is not critical so long as proper crosslinking is achieved.

Other agents such as viscosity modifiers, plasticizers, fillers, and other well-known additives may be incorporated where functionally desirable. For example, chelating agents may also be employed to sequester any metal contaminants present in the composition which would contribute to spurious curing prior to intended use.

According to the methods of the present invention, the microwave absorbable particles provide uniform heating and curing of microwave curable compositions that are otherwise substantially transparent to or non-absorbent of microwave energy. With the inventive methods, the temperature of the composition increases to and is maintained at a temperature, the Curie temperature of the particles, which is desirably a temperature sufficient to heat and cure but not degrade the composition, even when additional microwave energy is added to the composition. The unique nature of this method is superior over conventional heating techniques in that it is not only faster, but provides for a uniform heating and curing of thin or thick compositions without degrading any part of the compositions.

Useful microwave absorbable particles include ferromagnetic compounds, metal oxides, in general, ferrites, amorphous carbon containing compounds such as carbon black and graphite, and other materials which readily absorb microwave energy of the desired frequency.

Useful ferromagnetic compound particles include ferrites with spinel structure, hexagonal structure and nonlinear structure. Particularly useful ferromagnetic particles are spinel ferrites with the general formula of $M^{2+}Fe_2^{3+}O_4$, where M^{2+} is a divalent metal ion. Ferrite particles are especially useful for their inherent temperature limiting capabilities. The amorphous carbon compounds are preferred for their ability to produce controllable heating. Particles desirable have diameters from about 50 to about 1000 Angstroms and Curie temperature ranges from about 50°C to about 700°C. The microwave absorbable particles may be present in any amount to achieve the desired heating or curing effect. More specifically, the microwave absorbable particles are present in amounts of at least 10% by weight of the composition, and desirably about 10% to about 40% by weight of the total composition.

In the present invention, attention is given to the selection of microwave absorbable particles. The microwave absorbable particles are chosen for the frequency at which it absorbs radiation and for its Curie temperature. The Curie temperature is the temperature at which a material can no longer absorb microwave energy through its magnetic properties. The Curie temperature of the microwave absorbable particles is chosen so that it has the desired effect upon the curable resin component within which it is embedded. The microwave absorbable particles selected for use in the present invention may be chosen so that their Curie temperature is just above the fusion temperature of the curable resin component thereby allowing for the source of microwave energy of the system to be selectively used to cure the composition. The microwave absorbable particles are selected such that reaching their Curie temperature would not degrade the composition or otherwise deleteriously affect the desired properties of the cured composition.

When a magnetic particle reaches or exceeds a critical temperature, referred to as the Curie temperature, its magnetic permeability drops precipitously to a value approaching 1. The particle then loses much of its ability to respond to a magnetic field and heating is significantly diminished. When the temperature of the particle drops below the Curie point, the particle regains its magnetic properties and heating resumes. Therefore, when the temperature of the particle is less than the Curie temperature, the particle heats. When the temperature of the particle is greater than the Curie temperature, the particle essentially stops increasing in

temperature. Thus, the Curie point is a practical autoregulation means for preventing the host material from being overheated.

In contrast to the prior art, the present invention finds that curable compositions
5 containing high amounts of microwave absorbable particle, are not only practicable but also work well. For example, as evidenced in the examples, compositions containing microwave absorbable particles in an amount up to 40% by weight of the total composition cured extremely well.

10 Heat is generated throughout the region containing the microwave absorbable particles, permitting far greater control over the heating and curing of the compositions. Dispersing the particles evenly throughout the bulk of the composition facilitates uniform heating. Placement of the microwave absorbable particles, and the direction of microwave energy can be used to isolate the location of heat generation and curing. Thus, selective heating will result where the
15 particles are placed in higher concentrations in areas to be heated to a relatively greater extent. In either case, the temperature of articles loaded with the microwave absorbable particles is controllable by utilization of particles having a Curie temperature near the desired temperature.

It is contemplated that other frequencies outside the microwave range could be used so
20 long as it is able to cure the composition. For example, frequencies found in the radio frequency range, as are commonly known, are also useful. Useful frequencies include frequencies from about 10 KHz to about 100 GHz. Microwaves in the upper section of the frequency range are preferred due to the fact that they are easier to direct into a relatively narrow beam. The ability to collimate in focus such microwave energy is a particularly useful
25 feature in the context of this invention, as it allows the system operators to deliberately and remotely apply microwave energy to a particular location where it is desirable to heat the composition.

Other agents that may be added to the curable composition of the present invention
30 include catalysts, inhibitors, viscosity modifying agents, coloring agents, fluorescent agents and combinations thereof. Other agents may be used in an amount that imports the desired effect onto the composition and in particular in amounts from about 0.1% to about 40% by weight. Viscosity modifying agents may be used to desirably alter the viscosity of the uncured

composition. Particularly useful viscosity modifying agents include fumed silica, precipitated silica, water and combinations thereof. The silica may be either treated with cyclic polysiloxane, silazanes and combinations thereof. Useful viscosity modifying agents further include titanium dioxide, lithopone, zinc oxide, zirconium silicate, silica aerogel, iron oxide, diatomaceous earth, calcium carbonate, silazane treated silicas, glass fibers, magnesium oxide, chomic oxide, zirconium oxide, aluminum oxide, alpha quartz, calcined clay, carbon, graphite, cork, cotton, synthetic fibers, solid acetylinic acid and the like.

Stabilizers and inhibitors may also be employed to control and prevent premature peroxide decomposition and polymerization. Inhibitors and stabilizers incorporated into curing systems will prevent the compositions from curing at room temperature but won't prevent curing at elevated temperatures. Among those useful inhibitors include phenols such as hydroquinone and quinones as well as those selected from the class consisting of vinyl containing organocyclo tetrasiloxanes such as a methyl vinyl cyclotetrasiloxane, trialkylcyanurate, alkyl maleates and combinations thereof. Particularly useful inhibitors include hexamethyldisilazane (HMDS), amines, α -hydroxyacetylene and combinations thereof. The solvents methanol and ethanol and mixtures thereof may also be used as an inhibitor to suppress the reaction between curable component and the curing agents contained for almost indefinite periods at room temperature.

There are many applications where the methods and compositions of the present invention are useful. The present invention may be used for any application where microwave energy is used as the heating source. For example, the inventive curable compositions and the inventive curing methods work well for manufacturing basic materials such as gaskets and other preformed sealant devices.

The methods of the present invention permit formation of seals in a position remote to the source of the microwave energy. The inclusion of the microwave absorbable particles in a curable composition permits remote inspection of the seal. For example, in the application of the invention, a quantity of microwave absorbable particles is intermixed with the heat curable resin component that is in a putty-like form that is a flowable adhesive paste. This composition is applied between the surfaces to be bonded together. Then, the curable composition may be exposed to a collimated beam of microwaves radiated from the source of microwave radiation.

Such a beam may be easily and conveniently applied even when the curable composition is disposed behind a panel, so long as the panel is transparent to microwave radiation. The absorption of the beam of microwave energy by the microwave absorbable particles within the composition causes the curable composition to heat up to the Curie temperature of the ferromagnetic material and to cure the composition.

Additionally, the directability of the beam of microwaves from the microwave source, coupled with the fact that these microwaves easily penetrate through most non-metallic components, confers great utility of this particular embodiment of the system in creating joints or in repairing joints in compositions structures with microwave absorbable particles. Compositions and methods of the present invention allow joints and seals to be created without the application of unwanted heat to large portions of the composite structure being built, and further allows such heat to be selectively and remotely applied to portions of the resulting structure which are either physically inaccessible to the microwave source, or covered by microwave-transparent components such as panel.

When used as a bonding material, it may be useful for the inventive compositions to be in form of a tape that includes an adhesive on at least one side for affixing it to one side of the surface prior to the application of the microwave energy. The tape form of the inventive compositions can be used in the methods of the present invention.

EXAMPLES

Various compositions were prepared and tested for their suitability as a microwave curable composition. These compositions, as shown in Table 1 were prepared by combining the respective components as shown in table and uniformly mixing them together to form the compositions. Compositions typically included the curable resin component, microwave absorbable particles, curing agent, catalyst solution, inhibitors and viscosity modifiers. The amount of microwave absorbable particles ranged from about 6% to about 40% by weight. The test samples were prepared by placing the uncured inventive compositions onto a 3" long and ½-¾ diameter Ultem® slab. Ultem® (from General Electric, Fairfield, CT) is a polyetherimide plastic chosen for its microwave transparency and resistance to heat.

TABLE 1
INVENTIVE COMPOSITIONS (WEIGHT % OF TOTAL COMPOSITION)

Component	Composition 1	Composition 2	Composition 3	Composition 4	Composition 5	Composition 6
Heat Curable Resin Component						
Poly dimethylsiloxane-vinyl terminated (62,000 MW Vinyl Fluid)	29.22	29.07	26.57	32.28	25.56	20.62
Poly dimethylsiloxane, dimethyl, vinyl terminated (10,00 MW Vinyl Fluid)	32.83	32.67	27.31	33.18	27.30	21.20
Microwave Absorbable Particles						
Ferrite Particles ¹	15.00	14.93	22.74	6.08	22.73	40.00
Catalyst/Curing Agent						
Methyl hydrogen polysiloxane (25% pt catalyst solution)	2.70	3.2	3.00	3.65	3.00	2.33
Cyclo tri(vinyl methylsiloxane) + Pt (CO) ₂ Cl ₂	0.05	0.05	0.025	0.06	0.05	0.038
Inhibitors						
Hexamethyldisilazane (HMDS)			1.82	2.21	1.82	1.41
2-Phenyl-3-butyl-2-ol			0.18	0.22	0.18	0.14
Viscosity Modifiers						
Water			0.18	0.22	0.18	0.14
Silica	20.00	19.90	18.18	22.10	18.18	14.12
Solid acetylinic alcohol	0.2	0.2				

¹Manganese zinc ferrite (Steward Co.)

The ability of the compositions to cure over a period of time was observed and recorded in Table 2 and 3. The percent cured was measured by determining the amount of paste (uncured) versus the amount of solid material (cured). Also recorded in Table 2 and 3 is the hardness level of the composition as measured on the Shore Hardness scale. Table 3 describes curing speed and hardness of inventive compositions 4, 5 and 6 using the heat press curing method at 150°C and Table 2 describes curing speed and hardness of the inventive compositions cured using microwave energy. As shown in Table 2, inventive composition 6 with 40% microwave absorbing particles by weight of the total composition cured using microwave energy in less than 1 minute. Inventive composition 5, which had 22% by weight microwave absorbable particles, cured within 2 minutes and inventive composition 4, which has 6% by weight microwave absorbable particles, cured within 5 minutes. In contrast, as shown in Table 3, the inventive compositions 4,5 and 6 all cured at about 15 minutes using a heat press curing method at 150°C. Thus, curing times using microwave energy method are substantially faster than the curing times using the heat press curing method.

Additionally, Table 2 and 3 show that the curing process using heat press cure was less uniform than the microwave energy curing process. The inventive compositions cure more rapidly and uniformly with the microwave energy curing method than with the conventional curing methods.

The hardness values, as shown in Table 2 and 3, of the cured compositions using either curing method are all approximately equivalent. Thus, the amount of microwave absorbable particles does not adversely affect the hardness of the cured composition.

TABLE 2
Compositions Cured With Microwave Energy (High Energy)

Microwave Curing Time	Composition 4 (6% ferrite)		Composition 5 (22% ferrite)		Composition 6 (40% ferrite)	
	Observed Level of Cure	Hardness Shore A	Observed Level of Cure	Hardness Shore A	Observed Level of Cure	Hardness Shore A
1 min.	no cure	NA	Partially cured (surface/interior cured)	NA	100% cure	40
2 min.	no cure	NA	no cure at interface)	33	100% cure	40
4 min.	Partially cured (interface at edges uncured)	42	100% cure	35	100% cure	
5 min.		42	100% cure	36	100% cure	
6 min.	100% cure	--	100% cure	38	100% cure	
8 min.	100% cure	40	100% cure	37	100% cure	
10 min.	100% cure	--	100% cure	37	100% cure	
12 min.	100% cure	40	100% cure	38	100% cure	
15 min.	100% cure	41	100% cure	38	100% cure	

TABLE 3
C mpositions Cured With Heat Press Cure at 150° C

Curing Time	Composition 4 (6% ferrite)		Composition 5 (22% ferrite)		Composition 6 (40% ferrite)	
	Observed Level of Cure	Hardness Shore A	Observed Level of Cure	Hardness Shore A	Observed Level of Cure	Hardness Shore A
2 min.	no cure	NA	no cure	NA	no cure	NA
4 min.	no cure	NA	no cure	NA	no cure	NA
5 min.	no cure	NA	no cure	NA	no cure	NA
6 min.	partially cured (interior interfaced not cured)	NA	partially cured (interior & interface not cured)	NA	no cure	NA
8 min.	partially cured (interior & interface not cured)	NA	partially cured (interior & interface not cured)	NA	partially cured (interior & interface not cured)	NA
10 min.	partially cured (edges cured interior at center is soft)	NA	partially cured (edges cured, interior at center is soft)	NA	partially cured (edges cured, interior at center is soft)	NA
12 min.	partially cured (large central portion at interface uncured)	42	partially cured (small central portion at interface uncured)	42	partially cured (interior is soft)	NA
15 min.	cured	45	cured	48	cured (with some difficulty removing material)	50
20 min.	cured	47	cured	47	cured	47

Table 4, 5 and 6 compare the physical properties of the cured inventive composition 1, 2 and 3 produced by microwave energy and heat press curing methods. Compositions 1 and 2 have levels of microwave absorbable particles of about 15%, as shown in Table 1. It is evident from Table 4 and 5 that the microwave cured composition 1 and 2 have physical properties that are similar to those of the heat press cured composition 1 and 2. Upon further curing, the properties of the heat press cured compositions did not substantially change except for the elongation values and the compression set values decreased slightly. In contrast, the properties of the microwave cured compositions stayed substantially the same when they were further cured. Thus, further microwave curing of the inventive compositions does not enhance or degrade the physical properties of the inventive compositions.

As seen in Table 6, inventive composition 3 was microwave cured for 3.5 minutes followed with by a post cure by a heat press method for 16 hours at 150°C. The results from this table exemplify that further heat press curing of inventive composition 3 that was originally microwave cured does not substantially enhance or degrade the physical properties of the inventive composition. The post cured composition does show a slight increase in hardness, tensile strength, 50% modulus and fracture point, while a slight decrease was observed in the compression set and percent elongation values.

TABLE 4**Heat Curable Compositions Cured Using Conventional Heating Methods (Heat Press)**

	Composition 1		Composition 2	
	Heat Press Cure 1 hr. at 120°C. and 30 min. at 150°C.		Heat Press Cure 1 hr. at 120°C. and 30 min. at 150°C.	
	No Post Cure	Post Cured an Additional 18 hrs. at 150°C.	No Post Cure	Post Cured an Additional 18 hrs. at 150°C.
Hardness Shore A	43	44	51	51
Tensile Strength (psi)	419	490	449	481
50% Modulus (psi)	206	277	242	350
100% Modulus (psi)	347	340	392	--
% Elongation	130	110	116	79
Compression Set (% Original Deflection)	65	36	37	36

TABLE 5
Heat Curable Compositions Cured Using the Inventive Curing Method
(Microwave Energy)

	Composition 1		Composition 2	
	Microwaved Cured for 5 minutes at High Temperature		Microwaved Cured for 5 minutes at High Temperature	
	No Post Cure	Post Cured for 5 Minutes at High Temperature	No Post Cure	Post Cured in Microwave for 5 Minutes
Hardness Shore A	42	43	45	51
Tensile Strength (psi)	354	457	404	374
50% Modulus (psi)	146	186	178	220
100% Modulus (psi)	277	352	343	--
% Elongation	133	134	121	88
Compression Set (% Original Deflection)	59	60	46	45

TABLE 6
Microwave Cured/Post Heat Press Cured Composition

	Microwave cured for 3.5 min.	
	No post cure	Post cured by Heat Press for 16 hours at 150°C
Hardness Shore A	43	50
Tensile Strength (psi)	649	676
50% Modulus (psi)	251	306
100% Modulus (psi)	464	--
% Elongation	154	118
Compression Set (% Original Deflection)	42	30
Approx. Fracture Point (inch lbs.) (Sample thicknesses = 0.1")	76	83

From these examples it is evident that the inclusion of the microwave absorbing particles provides compositions that can be heated and cured by microwave energy with extraordinary control, yielding uniformly cured articles with faster curing times. The increased control over the application of heat to the composition will increase safety, reduce loss of heat to the surrounding environment and reduce manufacturing costs.

The invention being thus described, it will be obvious that the same may be varied in many ways. Such variations are not to be regarded as a departure from the spirit and scope of the invention and all modifications are intended to be included within the scope of the following claims.

WHAT IS CLAIMED IS:

1. A curable composition that is controllably curable by microwave energy comprising:

a) at least one heat curable resin component;

5 b) microwave absorbable particles in an amount of about 10% by weight of said composition; and

c) at least one curing agent for said heat curable resin component.

2. A composition according to claim 1 wherein said microwave absorbable particles
10 are in an amount sufficient to uniformly cure said composition.

3. A composition according to claim 1 wherein said microwave absorbable particles are in an amount of at least about 10% to about 40% by weight of said composition.

15 4. A composition according to claim 1 wherein said microwave absorbable particles are selected so that the Curie temperature of said particles is higher than the curing temperature of said heat curable resin component.

5. A composition according to claim 1 wherein said microwave absorbable particles
20 comprise a ferromagnetic compound.

6. A composition according to claim 1 wherein said microwave absorbable particles are selected from the group consisting of ferrite, amorphous carbon, carbon black, graphite and combinations thereof.

25 7. A composition according to claim 1 wherein said curable resin component is selected from the group consisting of acrylics, elastomeric synthetic rubbers, epoxies, polyesters, polyurethanes, polybutadienes, cyanate esters, bismoleimides, polyimides, phenolics, silicones, acrylic silicones, epoxy silicones and copolymers and combinations
30 thereof.

8. A composition according to claim 1 wherein said heat curable resin is an elastomeric composition.

9. A composition according to claim 1 wherein said heat curable resin is a heat curable silicone.

10. A composition according to claim 1 wherein said curing agent is an addition curing catalyst.

11. A composition according to claim 1 wherein said curing agent is selected from the group consisting of an amine-containing compound, an azo compound and a peroxide curing agent.

12. A composition according to claim 11 wherein said peroxide curing agent is selected from the group consisting of cumemhydroperoxide, methylethylketone hydroperoxide, percarbonates, diacyl peroxides, per-esters, per-acids and combinations thereof.

13. The composition accordingly to claim 1, further including a material selected from the group consisting of inhibitors, viscosity modifying agents, catalysts, coloring agents, fluorescent agents, and combinations thereof.

14. The compositions according to claim 13 wherein said viscosity modifying agent is selected from the group consisting of fumed silica, precipitated silica, titanium dioxide, lithopone, zinc oxide, zirconium silicate, silica aerogel, iron oxide, diatomaceous earth, calcium carbonate, silazane treated silicas, glass fibers, magnesium oxide, chromic oxide, zirconium oxide, aluminum oxide, alpha quartz, calcined clay, carbon, graphite, cork, cotton, synthetic fibers and combinations thereof.

15. The composition according to claim 13 wherein said viscosity modifying agent is in an amount from about 0.1 % to about 60 % of the total composition.

16. The compositions according to claim 13 wherein said catalyst comprises a platinum catalyst complex.

17. The compositions according to claim 13 wherein said catalyst is an amount from about 0.02 % to about 10.00 % of the total composition.

18. The compositions according to claim 13 wherein said inhibitor is selected from the group consisting of methylvinyl cyclotetrasiloxane, trialkylcyanurates, alkylmaleates and combinations thereof.

19. The compositions according to claim 13 wherein said inhibitor is selected from the group consisting of hexamethyldisilazane, amines, α - hydroxyacetylene and combinations thereof.

20. The composition according to claim 1 wherein said heat curable resin component is selected from the group consisting of monomers, oligomers, polymers and combinations thereof.

21. A method of controllably curing a composition comprising:

- a) selecting microwave absorbable particles having a desired Curie temperature;
- b) combining said microwave absorbable particles with a curing agent and a heat curable resin component which has a curing temperature lower than said Curie temperature, said resin being substantially transparent to microwave energy such that said particles being present in an amount of about 10% by weight of said composition; and
- c) applying a sufficient amount and wavelength of microwave energy to said composition to achieve the desired heating temperature.

22. A method according to claim 21 wherein said microwave absorbable particles are dispersed in an amount sufficient to cure said composition.

23. A method according to claim 21 wherein said composition is cured.

24. A method according to claim 21 wherein said microwave absorbable particles are dispersed only within selected portions of said heat curable resin component such that only said selected portions melt when said compositions are exposed to microwave energy.

5 25. A method according to claim 21 wherein the microwave absorbable particle comprises a ferromagnetic compound.

26. A method according to claim 21 wherein the microwave absorbable particle are selected from the group consisting of ferrite, amorphous carbon, carbon black, graphite and
10 combinations thereof.

27. A method according to claim 21 wherein said heat curable resin component is selected from the group consisting of acrylics, epoxies, polyesters, polyurethanes, polybutadienes, cyanate esters, bismoleimides, polyimides, phenolics, silicones and
15 combinations thereof.

28. A method according to claim 21 wherein said heat curable resin component is selected from the group consisting of monomers, oligomers, polymers and combinations thereof.
20

29. A method according to claim 21 wherein said microwave energy is in a range from about 10KHz to about 100 GHz.

30. A method according to claim 21 further comprising the step of applying said heat
25 curable resin component between two members to be joined and wherein the application of microwave energy melts said composition and joins said members.

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : Please See Extra Sheet.

US CL : 156/272.4; 522/006, 024, 029, 071, 074, 077, 083, 099, 148

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 156/272.4; 522/006, 024, 029, 071, 074, 077, 083, 099, 148

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

Please See Extra Sheet.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X --- Y	US 5,272,216 A (CLARK, JR. et al) 21 December 1993, abstract, col. 2, lines 24-68, col. 4, line 42-col. 5, line 19; col. 5, lines 30-42, claims 1-12.	1-9, 20-30 ----- 10-19
X --- Y	US 5,798,395 A (LAUF et al) 25 August 1998, abstract, col. 3, lines 2-26 and lines 42-58, examples 1 and 4, col. 11, lines 14-31.	1, 6-9, 11, 13-15, 20 ----- 2-5, 10, 12, 16-19, 21-30
X --- Y	US 5,061,736 A (TAKAHASHI et al.) 29 October 1991, abstract, col. 2, lines 18-51, col. 3, line 50-col. 5, line 13, col. 6, lines 48-66, col. 7, lines 1-17, example 1 and claims.	1-3, 5-17, 20 ----- 4, 18-19, 21-30



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:	*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
A document defining the general state of the art which is not considered to be of particular relevance	*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
E earlier document published on or after the international filing date	*Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
L document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	*Z* document member of the same patent family
O document referring to an oral disclosure, use, exhibition or other means	
P document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search

29 JANUARY 2001

Date of mailing of the international search report

21 FEB 2001

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X ---- Y	US 4,550,125 A (LEE et al.) 29 October 1985, col. 2, lines 44-51, col. 4, lines 40-43, col. 10, lines 23-30, col. 11, lines 6-23 and lines 42-65.	1-3, 6-10, 13-17, 20 ----- 4-5, 11-12, 18-19, 21-30
X ---- Y	US 4,980,384 A (TAKAHASHI et al.) 25 December 1990, abstract, col. 4, lines 21-66, col. 5, lines 16-39, and example 1.	1-2, 5-17, 20 ----- 3-4, 18-19, 21-30
Y	US 4,576,862A (LEE et al.) 18 March 1986, col. 2, lines 18-36, col. 5, line 55-col. 6, line 30.	1-30
X ---- Y	US 4,026,844 A (KITTLE et al.) 31 May 1977, abstract, col. 5, line 46-col. 6, line 10 and the example.	1-3, 6-10, 13-17, 20 ----- 4-5, 11-12, 18-19, 21-30
Y	US 5,438,081 A (LEWIS et al.) 01 August 1995, abstract, col. 1, line 54-col. 2, line 20, col. 3, lines 62-col. 4, line 7, col. 4, lines 28-35, col. 5, lines 26-30 and 48-59.	1-30

A. CLASSIFICATION OF SUBJECT MATTER:

IPC (7):

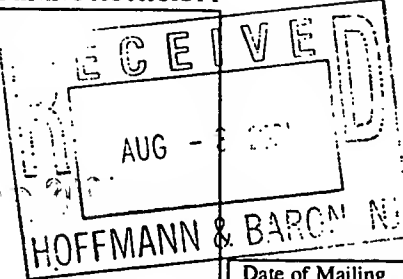
B32B 27/16, 27/18, 27/26, 31.28; C08J 3/28; C08K 3/22; C09J 5/06; C08F 2/44, 2/46, 4/04, 4/26

B. FIELDS SEARCHED

Electronic data bases consulted (Name of data base and where practicable terms used):

APS, DERWENT, EPO, JPO: microwave curing, microwave energy, Curie temperature, ferromagnetic, ferrite, carbon, graphite, elastomer, silicone, curing agent, peroxide, azo, amine, platinum catalyst, silica, titanium dioxide, filler

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PCT

AUG 6 2001

WRITTEN OPINION

(PCT Rule 66)

HOFFMANN & BARON, LLP

PTO/PCT Rec'd

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International application No. PCT/US00/29134		REPLY DUE within 2 months/days from the above date of mailing
International filing date (day/month/year) 20 October 2000 (20.10.2000)	Priority date (day/month/year) 20 OCTOBER 1999	
International Patent Classification (IPC) or both national classification and IPC IPC(7): B32B 27/16, 27/18, 27/26, 31/28; C08J 3/28; C08K 3/22; C09J 5/06; C08F 2/44, 2/46, 4/04, 4/26. and US Cl.: 156/272.4; 522/ 006, 024, 029, 071, 074, 077, 083, 099, 148.		
Applicant LOCTITE CORPORATION		

- This written opinion is the first (first, etc.) drawn by this International Preliminary Examining Authority.
- This opinion contains indications relating to the following items:
 - ☒ Basis of the opinion
 - ☐ Priority
 - ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
 - ☐ Lack of unity of invention
 - ☒ Reasoned statement under Rule 66.2 (a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
 - ☐ Certain documents cited
 - ☐ Certain defects in the international application
 - ☒ Certain observations on the international application
- The applicant is hereby invited to reply to this opinion.

When? See the time limit indicated above. ~~The applicant may, before the expiration of that time limit, request this Authority to grant an extension. See rule 66.2(d).~~

How? By submitting a written reply, accompanied, where appropriate, by amendments, according to Rule 66.3. For the form and the language of the amendments, see Rules 66.8 and 66.9.

Also For an additional opportunity to submit amendments, see Rule 66.4.
For the examiner's obligation to consider amendments and/or arguments, see Rule 66.4 bis.
For an informal communication with the examiner, see Rule 66.6

If no reply is filed, the international preliminary examination report will be established on the basis of this opinion.
- The final date by which the international preliminary examination report must be established according to Rule 69.2 is: 20 February 2001 (20.02.2001).

Name and mailing address of the IPEA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703)305-3230	Authorized officer Susan W Berman Telephone No. 703 308 0661
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I. Basis of the opinion

1. With regard to the elements of the international application:*

- ☒ the international application as originally filed
- ☒ the description:
pages 1-26, as originally filed
pages NONE, filed with the demand
pages NONE, filed with the letter of _____.
- ☒ the claims:
pages 27-30, as originally filed
pages NONE, as amended (together with any statement) under Article 19
pages NONE, filed with the demand
pages NONE, filed with the letter of _____.
- ☒ the drawings:
pages NONE, as originally filed
pages NONE, filed with the demand
pages NONE, filed with the letter of _____.
- ☐ the sequence listing part of the description:
pages NONE, as originally filed
pages NONE, filed with the demand
pages NONE, filed with the letter of _____.

2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item. These elements were available or furnished to this Authority in the following language _____ which is:

- ☐ the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the written opinion was drawn on the basis of the sequence listing:

- ☐ contained in the international application in printed form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. ☒ The amendments have resulted in the cancellation of:

- ☒ the description, pages NONE
- ☒ the claims, Nos. NONE
- ☒ the drawings, sheets/fig NONR

5. ☐ This opinion has been drawn as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).

* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this opinion as "originally filed."

V. Reasoned statement under Article 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. STATEMENT

Novelty (N)	Claims <u>10-19</u>	YES
	Claims <u>1-9,20-30</u>	NO
Inventive Step (IS)	Claims <u>NONE</u>	YES
	Claims <u>1-30</u>	NO
Industrial Applicability (IA)	Claims <u>1-30</u>	YES
	Claims <u>NONE</u>	NO

2. CITATIONS AND EXPLANATIONS

Claims 1-9 and 20-30 lack novelty under PCT Article 33(2) as being anticipated by CLARK, JR. ET AL.. CLARK, JR. ET AL. disclose curable compositions comprising between 0.1 to 10 percent by weight of microwaveable absorbable particles of the resulting polymeric composite. The particles, such as ferrite, are selected so that the Curie temperature of the particles is higher than the curing temperature of the curable resin. Thermosetting polymers, including silicones, can be hardened. The method taught includes directing microwave energy to a selected portion of the polymeric composite. The claim recitation "about 10% by weight of said composition" is considered to include 10 % by weight of the resulting polymeric composite as taught by CLARK, JR. ET AL.

Claims 1-17 and 20-30 lack an inventive step under PCT Article 33(3) as being obvious over CLARK, JR. ET AL. in view of TAKAHASHI ET AL. CLARK, JR. ET AL teach microwave curable compositions comprising between 0.1 to 10 % by weight ferromagnetic particles and thermosetting polymers. TAKAHASHI ET AL teach adding dielectric materials, such as ferrites, in amounts ranging from 5 to 100 parts by weight of an organopolysiloxane. TAKAHASHI ET AL teach addition curing an organopolysiloxane composition comprising finely divided silica filler, a peroxide crosslinking agent and a platinum hydrosilation catalyst with microwave energy to provide cured and foamed silicone rubber. It would not have involved an inventive step to employ the organopolysiloxane compositions taught by TAKAHASHI ET AL as the thermosetting silicones suggested by CLARK, JR. ET AL. One skilled in the art at the time of the invention would have been motivated by an expectation of providing a cured and foamed silicone rubber using particles selected by Curie temperature, as taught by CLARK, JR. ET AL., and adding an amount of 10% or greater dielectric material to ensure sufficient absorption of microwave energy, as taught by TAKAHASHI ET AL.

Claims 1-10 and 13-30 lack an inventive step under PCT Article 33(3) as being obvious over CLARK, JR. ET AL. in view of KITTLE ET AL. See the discussion of teachings of CLARK, JR. ET AL. set forth above. KITTLE ET AL teach also adding from 0.25 to 30 parts by weight microwave sensible material, such as carbon black and metal oxides, to polydiorganosiloxane compositions to be cured by exposure to microwave energy. KITTLE ET AL teach also adding an acetylenic alcohol inhibitor to the polydiorganosiloxane compositions. It would not have involved an inventive step to employ the polydiorganosiloxane compositions taught by KITTLE ET AL as the thermosetting silicones suggested by CLARK, JR. ET AL. One skilled in the art at the time of the invention would have been motivated by an expectation of providing a cured and foamed silicone rubber using particles selected by Curie temperature, as taught by CLARK, JR. ET AL., and adding an amount of 0.25 to 30% by weight microwave sensible material, as taught by KITTLE ET AL.

Claims 1-30 meet the criteria set out in PCT Article 33(4) because the composition and method are useful for curing polymeric composites.

----- NEW CITATIONS -----

VIII. Certain observations on international application

The following observations on the clarity of the claims, description, and drawings or on the questions whether the claims are fully supported by the description, are made:

Claims 1-3 are objected to under PCT Rule 66.2(a)(v) as lacking clarity under PCT Article 6 because claim 1 is indefinite for the following reason(s): Claim 1 recites microwave absorbable particles in "an amount of about 10% by weight of said composition". Claim 2 recites "an amount sufficient to uniformly cure said compositions" and claim 3 recites "at least about 10% to about 40% by weight of said composition". It is not clear what amount of microwave absorbable particles is intended to be claimed since claim 1 limits the amount to 10% by weight but claims 2 and 3 recite amounts different than 10% by weight.

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CHAPTER II

DEMAND

under Article 31 of the Patent Cooperation Treaty:
The undersigned requests that the international application specified below be the subject of
international preliminary examination according to the Patent Cooperation Treaty and
hereby elects all eligible States (except where otherwise indicated).

For International Preliminary Examining Authority use only		
Identification of IPEA		Date of receipt of DEMAND
Box No. I IDENTIFICATION OF THE INTERNATIONAL APPLICATION		Applicant's or agent's file reference 500-46 PCT
International application No. PCT/US00/29134	International filing date (day/month/year) 20 OCTOBER 2000 (20.10.00)	(Earliest) Priority date (day/month/year) 20 OCTOBER 1999 (20.10.99)
Title of invention MICROWAVE CURABLE COMPOSITIONS		
Box No. II APPLICANT(S)		
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) LOCTITE CORPORATION 1001 Trout Brook Crossing Rocky Hill, Connecticut 06067 US		Telephone No.: 860-571-5100
		Facsimile No.: 860-571-5028
		Teleprinter No.:
State (that is, country) of nationality: US		State (that is, country) of residence: US
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) ARGAZZI, Dennis J. 13 Woodland Drive Centerbrook, CT 60409 US		
State (that is, country) of nationality: US		State (that is, country) of residence: US
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) HAAS, Hans E. 44 Meetinghouse Lane Westbrook, CT 06498 US		
State (that is, country) of nationality: US		State (that is, country) of residence: US
<input checked="" type="checkbox"/> Further applicants are indicated on a continuation sheet.		

Continuation of Box No. II APPLICANT(S)

If none of the following sub-boxes is used, this sheet is not to be included in the demand.

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

LIM, Thomas Fay-oy
128 Route 148
Killingworth, CT 06419
US

State *(that is, country)* of nationality:
US

State *(that is, country)* of residence:
US

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

State *(that is, country)* of nationality:

State *(that is, country)* of residence:

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

State *(that is, country)* of nationality:

State *(that is, country)* of residence:

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

State *(that is, country)* of nationality:

State *(that is, country)* of residence:

☐

Further applicants are indicated on another continuation sheet.

Box No. III AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE

The following person is ☒ agent ☐ common representative
 and ☒ has been appointed earlier and represents the applicant(s) also for international preliminary examination.
☐ is hereby appointed and any earlier appointment of (an) agent(s) /common representative is hereby revoked.
☐ is hereby appointed, specifically for the procedure before the International Preliminary Examining Authority, in addition to the agent(s)/common representative appointed earlier.

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

SCOLA, Daniel A., Jr., Esq.
 HOFFMANN & BARON, LLP
 6900 Jericho Turpike
 Syosset, New York 11791
 USA

Telephone No.:
 (973) 331-1700

Facsimile No.:
 (973) 31-1717

Teleprinter No.:

☐ Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

Box No. IV BASIS FOR INTERNATIONAL PRELIMINARY EXAMINATION**Statement concerning amendments:***

1. The applicant wishes the international preliminary examination to start on the basis of:

☒ the international application as originally filed.

the description ☒ as originally filed
☐ as amended under Article 34

the claims ☒ as originally filed
☐ as amended under Article 19 (together with any accompanying statement)
☐ as amended under Article 34

the drawings ☐ as originally filed
☐ as amended under Article 34

2. ☐ The applicant wishes any amendment to the claims under Article 19 to be considered as reversed.

3. ☐ The applicant wishes the start of the international preliminary examination to be postponed until the expiration of 20 months from the priority date unless the International Preliminary Examining Authority receives a copy of any amendments made under Article 19 or a notice from the applicant that he does not wish to make such amendments (Rule 69.1(d)). *(This check-box may be marked only where the time limit under Article 19 has not yet expired.)*

* Where no check-box is marked, international preliminary examination will start on the basis of the international application as originally filed or, where a copy of amendments to the claims under Article 19 and/or amendments of the international application under Article 34 are received by the International Preliminary Examining Authority before it has begun to draw up a written opinion or the international preliminary examination report, as so amended.

Language for the purposes of international preliminary examination: English

☒ which is the language in which the international application was filed.
☐ which is the language of a translation furnished for the purposes of international search.
☐ which is the language of publication of the international application.
☐ which is the language of the translation (to be) furnished for the purposes of international preliminary examination.

Box No. V ELECTION OF STATES

The applicant hereby elects all eligible States *(that is, all States which have been designated and which are bound by Chapter II of the PCT)*

excluding the following States which the applicant wishes not to elect:

Box No. VI CHECK LIST

The demand is accompanied by the following elements, in the language referred to in Box No. IV, for the purposes of international preliminary examination:

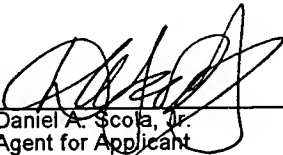
			For International Preliminary Examining Authority use only	
			received	not received
1.	translation of international application	sheets	<input type="checkbox"/>	<input type="checkbox"/>
2.	amendments under Article 34	sheets	<input type="checkbox"/>	<input type="checkbox"/>
3.	copy (or where required, translation) of amendments under Article 19	sheets	<input type="checkbox"/>	<input type="checkbox"/>
4.	copy (or, where required, translation) of statement under Article 19	sheets	<input type="checkbox"/>	<input type="checkbox"/>
5.	letter	sheets	<input type="checkbox"/>	<input type="checkbox"/>
6.	other (<i>specify</i>)	sheets	<input type="checkbox"/>	<input type="checkbox"/>

The demand is also accompanied by the item(s) marked below:

- | | |
|--|---|
| 1. <input checked="" type="checkbox"/> fee calculation sheet | 4. <input type="checkbox"/> statement explaining lack of signature |
| 2. <input type="checkbox"/> separate signed power of attorney | 5. <input type="checkbox"/> nucleotide and or amino acid sequence listing in computer readable form |
| 3. <input type="checkbox"/> copy of general power of attorney; reference number, if any: | 6. <input checked="" type="checkbox"/> other (<i>specify</i>): Transmittal Letter |

Box No. VII SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the demand).



Daniel A. Scola, Jr.
Agent for Applicant

For International Preliminary Examining Authority use only

1. Date of actual receipt of DEMAND:

2. Adjusted date of receipt of demand due to CORRECTIONS under Rule 60.1(b):

3. ☐ The date of receipt of the demand is AFTER the expiration of 19 months from the priority date and item 4 or 5, below, does not apply.

☐ The applicant has been informed accordingly.

4. ☐ The date of receipt of the demand is WITHIN the period of 19 months from the priority date as extended by virtue of Rule 80.5.

5. ☐ Although the date of receipt of the demand is after the expiration of 19 months from the priority date, the delay in arrival is EXCUSED pursuant to Rule 82.

For International Bureau use only

Demand received from IPEA on:

PCT

FEE CALCULATION SHEET

Annex to the Demand for international preliminary examination

International application No. PCT/US00/29134	For International Preliminary Examining Authority use only	
Applicant's or agent's file reference 500-46 PCT	Date stamp of the IPEA	
Applicant LOCTITE CORPORATION		
Calculation of prescribed fees 1. Preliminary examination fee 750.00 P 2. Handling fee (<i>Applicants from certain States are entitled to a reduction of 75% of the handling fee. Where the applicant is (or all applicants are) so entitled, the amount to be entered at H is 25% of the handling fee.</i>) 137.00 H 3. Total of prescribed fees Add the amounts entered at P and H and enter total in the TOTAL box 887.00 <div style="text-align: right; border: 1px solid black; padding: 2px 10px; margin-top: 2px;">TOTAL</div>		
Mode of Payment <div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <input type="checkbox"/> authorization to charge deposit account with the IPEA (see below) </div> <div style="width: 45%;"> <input type="checkbox"/> cash </div> </div> <div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <input checked="" type="checkbox"/> cheque </div> <div style="width: 45%;"> <input type="checkbox"/> revenue stamps </div> </div> <div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <input type="checkbox"/> postal money order </div> <div style="width: 45%;"> <input type="checkbox"/> coupons </div> </div> <div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <input type="checkbox"/> bank draft </div> <div style="width: 45%;"> <input type="checkbox"/> other (specify): </div> </div>		
Deposit Account Authorization (<i>this mode of payment may not be available at all IPEAs</i>) The IPEA/ <u>US</u> <input type="checkbox"/> is hereby authorized to charge the total fees indicated above to my deposit account. <input type="checkbox"/> (<i>this check-box may be marked only if the conditions for deposit accounts of the IPEA so permit</i>) is hereby authorized to charge any deficiency or credit any overpayment in the total fees indicated above to my deposit account.		
08-2461	25 April 2001	
Deposit Account Number	Date (day/month/year)	Signature

To: DANIEL A. SCOLA, JR.
HOFFMANN & BARON, LLP
6900 JERICHO TURNPIKE
SYOSSET NY 11791

PTO/PCT Rec'd 13 MAR 2002

PCT

NOTIFICATION OF TRANSMITTAL OF
THE INTERNATIONAL SEARCH REPORT
OR THE DECLARATION

(PCT Rule 44.1)

Date of Mailing (day/month/year) 21 FEB 2001	
Applicant's or agent's file reference 500-46 PCT-1	FOR FURTHER ACTION See paragraphs 1 and 4 below
International application No. PCT/US00/29134	International filing date (day/month/year) 20 OCTOBER 2000
Applicant LOCTITE CORPORATION	

1. ☒ The applicant is hereby notified that the international search report has been established and is transmitted herewith.

Filing of amendments and statement under Article 19:
The applicant is entitled, if he so wishes, to amend the claims of the international application (see Rule 46):

When? The time limit for filing such amendments is normally 2 months from the date of transmittal of the international search report; however, for more details, see the notes on the accompanying sheet.

Where? Directly to the International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland
Facsimile No.: (41-22) 740.14.35

For more detailed instructions, see the notes on the accompanying sheet.

2. ☐ The applicant is hereby notified that no international search report will be established and that the declaration under Article 17(2)(a) to that effect is transmitted herewith.

3. ☐ With regard to the protest against payment of (an) additional fee(s) under Rule 40.2, the applicant is notified that:

☐ the protest together with the decision thereon has been transmitted to the International Bureau together with the applicant's request to forward the texts of both the protest and the decision thereon to the designated Offices.

☐ no decision has been made yet on the protest; the applicant will be notified as soon as a decision is made.

4. **Further action(s):** The applicant is reminded of the following:

Shortly after 18 months from the priority date, the international application will be published by the International Bureau. If the applicant wishes to avoid or postpone publication, a notice of withdrawal of the international application, or of the priority claim, must reach the International Bureau as provided in rules 90 bis 1 and 90 bis 3, respectively, before the completion of the technical preparations for international publication.

Within 19 months from the priority date, a demand for international preliminary examination must be filed if the applicant wishes to postpone the entry into the national phase until 30 months from the priority date (in some Offices even later).

Within 20 months from the priority date, the applicant must perform the prescribed acts for entry into the national phase before all designated Offices which have not been elected in the demand or in a later election within 19 months from the priority date or could not be elected because they are not bound by Chapter II.

Name and mailing address of the ISA/US: Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231	Authorized officer SUSAN W. BERMAN <i>Susan W. Berman</i>
Facsimile No. (703) 305-3230	Telephone No. (703) 308-0651

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference 500-46 PCT-1	FOR FURTHER ACTION	see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.
International application No. PCT/US00/29134	International filing date (day/month/year) 20 OCTOBER 2000	(Earliest) Priority Date (day/month/year) 20 OCTOBER 1999
Applicant LOCTITE CORPORATION		

This international search report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This international search report consists of a total of 4 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

- a. With regard to the language, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.
- ☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).
- b. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international search was carried out on the basis of the sequence listing:
- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.
2. ☐ Certain claims were found unsearchable (See Box I).
3. ☐ Unity of invention is lacking (See Box II).
4. With regard to the title,
- ☒ the text is approved as submitted by the applicant.
- ☐ the text has been established by this Authority to read as follows:
5. With regard to the abstract,
- ☒ the text is approved as submitted by the applicant.
- ☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.
6. The figure of the drawings to be published with the abstract is Figure No. _____
- ☐ as suggested by the applicant.
- ☐ because the applicant failed to suggest a figure.
- ☐ because this figure better characterizes the invention.
- ☐ None of the figures.

A. CLASSIFICATION OF SUBJECT MATTER IPC(7) : Please See Extra Sheet. US CL : 156/272.4; 522/006, 024, 029, 071, 074, 077, 083, 099, 148 According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) U.S. : 156/272.4; 522/006, 024, 029, 071, 074, 077, 083, 099, 148 Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched NONE Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) Please See Extra Sheet.		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X --- Y	US 5,272,216 A (CLARK, JR. et al) 21 December 1993, abstract, col. 2, lines 24-68, col. 4, line 42-col. 5, line 19; col. 5, lines 30-42, claims 1-12.	1-9, 20-30 ----- 10-19
X --- Y	US 5,798,395 A (LAUF et al) 25 August 1998, abstract, col. 3, lines 2-26 and lines 42-58, examples 1 and 4, col. 11, lines 14-31.	1, 6-9, 11, 13-15, 20 ----- 2-5, 10, 12, 16-19, 21-30
X --- Y	US 5,061,736 A (TAKAHASHI et al.) 29 October 1991, abstract, col. 2, lines 18-51, col. 3, line 50-col. 5, line 13, col. 6, lines 48-66, col. 7, lines 1-17, example 1 and claims.	1-3, 5-17, 20 ----- 4, 18-19, 21-30
<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C. <input type="checkbox"/> See patent family annex.		
* *A* *E* *L* *O* *P*	Special categories of cited documents: document defining the general state of the art which is not considered to be of particular relevance earlier document published on or after the international filing date document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) document referring to an oral disclosure, use, exhibition or other means document published prior to the international filing date but later than the priority date claimed	*T* *X* *Y* *G* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art document member of the same patent family
Date of the actual completion of the international search 29 JANUARY 2001		Date of mailing of the international search report 24 FEB 2001
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230		Authorized officer SUSAN W. BERMAN <i>Susan W. Berman</i> Telephone No. (703) 308-0651

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X ---- Y	US 4,550,125 A (LEE et al.) 29 October 1985, col. 2, lines 44-51, col. 4, lines 40-43, col. 10, lines 23-30, col. 11, lines 6-23 and lines 42-65.	1-3, 6-10, 13-17, 20 ----- 4-5, 11-12, 18-19, 21-30
X ---- Y	US 4,980,384 A (TAKAHASHI et al.) 25 December 1990, abstract, col. 4, lines 21-66, col. 5, lines 16-39, and example 1.	1-2, 5-17, 20 ----- 3-4, 18-19, 21-30
Y	US 4,576,862A (LEE et al.) 18 March 1986, col. 2, lines 18-36, col. 5, line 55-col. 6, line 30.	1-30
X ---- Y	US 4,026,844 A (KITTLE et al.) 31 May 1977, abstract, col. 5, line 46-col. 6, line 10 and the example.	1-3, 6-10, 13-17, 20 ----- 4-5, 11-12, 18-19, 21-30
Y	US 5,438,081 A (LEWIS et al.) 01 August 1995, abstract, col. 1, line 54-col. 2, line 20, col. 3, lines 62-col. 4, line 7, col. 4, lines 28-35, col. 5, lines 26-30 and 48-59.	1-30

A. CLASSIFICATION OF SUBJECT MATTER:

IPC (7):

B32B 27/16, 27/18, 27/26, 31,28; C08J 3/28; C08K 3/22; C09J 5/06; C08F 2/44, 2/46, 4/04, 4/26

B. FIELDS SEARCHED

Electronic data bases consulted (Name of data base and where practicable terms used):

APS, DERWENT, EPO, JPO: microwave curing, microwave energy, Curie temperature, ferromagnetic, ferrite, carbon, graphite, elastomer, silicone, curing agent, peroxide, azo, amine, platinum catalyst, silica, titanium dioxide, filler

PCT REQUEST

500-46 PCT-1

Original (for SUBMISSION) - printed on 20.10.2000 05:20:56 PM

0	For receiving Office use only	
0-1	International Application No.	
0-2	International Filing Date	
0-3	Name of receiving Office and "PCT International Application"	
0-4	Form - PCT/RO/101 PCT Request	
0-4-1	Prepared using	PCT-EASY Version 2.91 (updated 10.05.2000)
0-5	Petition The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty	
0-6	Receiving Office (specified by the applicant)	United States Patent and Trademark Office (USPTO) (RO/US)
0-7	Applicant's or agent's file reference	500-46 PCT-1
I	Title of invention	MICROWAVE CURABLE COMPOSITIONS
II	Applicant	
II-1	This person is:	applicant only
II-2	Applicant for	all designated States except US
II-4	Name	LOCTITE CORPORATION
II-5	Address:	1001 Trout Brook Crossing Rocky Hill, CT 06067 United States of America
II-6	State of nationality	US
II-7	State of residence	US
II-8	Telephone No.	860-571-5001
II-9	Facsimile No.	860-571-5028
III-1	Applicant and/or inventor	
III-1-1	This person is:	applicant and inventor
III-1-2	Applicant for	US only
III-1-4	Name (LAST, First)	ARGAZZI, Dennis, J.
III-1-5	Address:	13 Woodland Drive Centerbrook, CT 06409 United States of America
III-1-6	State of nationality	US
III-1-7	State of residence	US

PCT REQUEST

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Original (for SUBMISSION) - printed on 20.10.2000 05:20:56 PM

III-2	Applicant and/or inventor	
III-2-1	This person is:	applicant and inventor
III-2-2	Applicant for	US only
III-2-4	Name (LAST, First)	HAAS, Hans, E.
III-2-5	Address:	44 Meetinghouse Lane Westbrook, CT 06498 United States of America
III-2-6	State of nationality	US
III-2-7	State of residence	US
III-3	Applicant and/or inventor	
III-3-1	This person is:	applicant and inventor
III-3-2	Applicant for	US only
III-3-4	Name (LAST, First)	LIM, Thomas, Fay-oy
III-3-5	Address:	128 Route 148 Killingworth, CT 06419 United States of America
III-3-6	State of nationality	US
III-3-7	State of residence	US
IV-1	Agent or common representative; or address for correspondence The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as:	agent
IV-1-1	Name (LAST, First)	SCOLA, Daniel, A., Jr.
IV-1-2	Address:	HOFFMANN & BARON, LLP 6900 Jericho Turnpike Syosset, NY 11791 United States of America
IV-1-3	Telephone No.	973-331-1700
IV-1-4	Facsimile No.	973-331-1717
IV-1-5	e-mail	dscola@hoffmannbaron.com
IV-2	Additional agent(s)	additional agent(s) with same address as first named agent
IV-2-1	Name(s)	ABBRUZZESE, Salvatore, J.; BARON, Mark, E.; MERKEL, Kellyanne; SOPKO, John, S.; JACOBSEN, Barry, H.; SZAKIEL, Gloria, K.; CUSICK, Clinton, J.; WARRICK, Christina, L.; HOFFMANN, Charles, R.; BARON, Ronald, J.; BODNER, Gerald, T.; SACK, Alan, M.; KAMMER, A., Thomas; SCHROEDER, R., Glenn; HENNEBERGER, Glenn, T.; FEIT, Irving, N.; BENNETT, Anthony, E.; BACHMANN, Gregory, W.; ZUSCHLAG, Steven, T.; SIPOS, Susan, A.; MCDERMOTT, Kevin, E.; MORRISS, Robert, C.; TURNER, Roderick, S., W.; HARRINGTON, James, F.; ANILINOIS, Algis; HOLMES, Justin, K.

PCT REQUEST

500-46 PCT-1

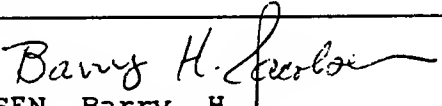
Original (for SUBMISSION) - printed on 20.10.2000 05:20:56 PM

IV-3	Additional agent(s)	agent
IV-3-1	Name (LAST, First)	BAUMAN, Steven, C.
IV-3-2	Address:	1001 Trout Brook Crossing Rocky Hill, CT 06067 United States of America
IV-3-3	Telephone No.	860-571-5001
IV-3-4	Facsimile No.	860-571-5028
V	Designation of States	
V-1	Regional Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)	AP: GH GM KE LS MW MZ SD SL SZ TZ UG ZW and any other State which is a Contracting State of the Harare Protocol and of the PCT EA: AM AZ BY KG KZ MD RU TJ TM and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT EP: AT BE CH&LI CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE and any other State which is a Contracting State of the European Patent Convention and of the PCT OA: BF BJ CF CG CI CM GA GN GW ML MR NE SN TD TG and any other State which is a member State of OAPI and a Contracting State of the PCT
V-2	National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)	AE AG AL AM AT AU AZ BA BB BG BR BY BZ CA CH&LI CN CR CU CZ DE DK DM DZ EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT TZ UA UG US UZ VN YU ZA ZW
V-5	Precautionary Designation Statement In addition to the designations made under items V-1, V-2 and V-3, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except any designation(s) of the State(s) indicated under item V-6 below. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit.	
V-6	Exclusion(s) from precautionary designations	NONE
VI-1	Priority claim of earlier national application	
VI-1-1	Filing date	20 October 1999 (20.10.1999)
VI-1-2	Number	60/160,577
VI-1-3	Country	US

PCT REQUEST

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VI-2	Priority document request The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) identified above as item(s):	VI-1	
VII-1	International Searching Authority Chosen	United States Patent and Trademark Office (USPTO) (ISA/US)	
VIII	Check list	number of sheets	electronic file(s) attached
VIII-1	Request	5	-
VIII-2	Description	26	-
VIII-3	Claims	4	-
VIII-4	Abstract	1	document3.txt
VIII-5	Drawings	0	-
VIII-7	TOTAL	36	
	Accompanying items	paper document(s) attached	electronic file(s) attached
VIII-8	Fee calculation sheet	✓	-
VIII-10	Copy of general power of attorney	reference no. 500-46 PCT-1	-
VIII-16	PCT-EASY diskette	-	diskette
VIII-17	Other (specified):	Authorization Sheet For Steven C. Bauman	-
VIII-17	Other (specified):	Assignment	-
VIII-17	Other (specified):	Declaration and Power of Attorney	-
VIII-18	Figure of the drawings which should accompany the abstract		
VIII-19	Language of filing of the international application	English	
IX-1	Signature of applicant or agent		
IX-1-1	Name (LAST, First)	JACOBSEN, Barry, H.	

FOR RECEIVING OFFICE USE ONLY

10-1	Date of actual receipt of the purported international application	
10-2	Drawings:	
10-2-1	Received	
10-2-2	Not received	
10-3	Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application	
10-4	Date of timely receipt of the required corrections under PCT Article 11(2)	
10-5	International Searching Authority	ISA/US
10-6	Transmittal of search copy delayed until search fee is paid	

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FOR INTERNATIONAL BUREAU USE ONLY

11-1	Date of receipt of the record copy by the International Bureau	
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PCT (ANNEX - FEE CALCULATION SHEET)

500-46 PCT-1

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(This sheet is not part of and does not count as a sheet of the international application)

0	For receiving Office use only		
0-1	International Application No.		
0-2	Date stamp of the receiving Office		
0-4	Form - PCT/RO/101 (Annex)		
0-4-1	PCT Fee Calculation Sheet Prepared using	PCT-EASY Version 2.91 (updated 10.05.2000)	
0-9	Applicant's or agent's file reference	500-46 PCT-1	
2	Applicant	LOCTITE CORPORATION, et al.	
12	Calculation of prescribed fees	fee amount/multiplier	total amounts (USD)
12-1	Transmittal fee T	⇒	240
12-2	Search fee S	⇒	700
12-3	International fee		
	Basic fee (first 30 sheets) b1	427	
12-4	Remaining sheets	6	
12-5	Additional amount (X)	10	
12-6	Total additional amount b2	60	
12-7	b1 + b2 = B	487	
12-8	Designation fees		
	Number of designations contained in international application	87	
12-9	Number of designation fees payable (maximum 8)	8	
12-10	Amount of designation fee (X)	92	
12-11	Total designation fees D	736	
12-12	PCT-EASY fee reduction R	-132	
12-13	Total International fee (B+D-R) I	⇒	1,091
12-14	Fee for priority document		
	Number of priority documents requested	1	
12-15	Fee per document (X)	15	
12-16	Total priority document fee P	⇒	15
12-17	TOTAL FEES PAYABLE (T+S+I+P)	⇒	2,046
12-19	Mode of payment	cheque	
12-20	Deposit account instructions		
	The receiving Office:	United States Patent and Trademark Office (USPTO) (RO/US)	
12-20-2	is hereby authorized to charge any deficiency or credit any over-payment in the total fees indicated above to my deposit account	✓	
12-20-3	is hereby authorized to charge the fee for preparation and transmittal of the priority document to the International Bureau of WIPO to my deposit account	✓	
12-21	Deposit account No.	08-2461	

PCT (ANNEX - FEE CALCULATION SHEET)

500-46 PCT-1

Original (for SUBMISSION) - printed on 20.10.2000 05:20:56 PM

12-22	Date	20 October 2000 (20.10.2000)
12-23	Name and signature	JACOBSEN, Barry, H. <i>Barry H. Jacobsen</i>

VALIDATION LOG AND REMARKS

13-2-6	Validation messages Contents	Green? The international application contains no drawings. Please verify.
13-2-7	Validation messages Fees	Green? Please confirm that fee schedule utilized is the latest available

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:
DANIEL A. SCOLA, JR.
HOFFMANN & BARON, LLP
6900 JERICHO TURNPIKE
SYOSSET, NY 11791

PCT

NOTIFICATION OF TRANSMITTAL OF
INTERNATIONAL PRELIMINARY
EXAMINATION REPORT

(PCT Rule 71.1)

Date of Mailing
(day/month/year)

05 APR 2002

Applicant's or agent's file reference

500-46 PCT-1

IMPORTANT NOTIFICATION

International application No.

International filing date (day/month/year)

Priority date (day/month/year)

PCT/US00/29134

20 October 2000 (20.10.2000)

20 October 1999 (20.10.1999)

Applicant

LOCTITE CORPORATION

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.
4. **REMINDER**

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices)(Article 39(1))(see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/US

Commissioner of Patents and Trademarks
Box PCT
Washington, D.C. 20231

Facsimile No. (703)305-3230

Authorized officer
Susan W Berman
Susan W Berman

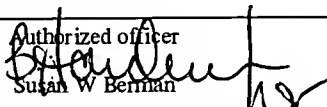
Telephone No. 703 308 0661

Form PCT/IPEA/416 (July 1992)

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 500-46 PCT-1	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/US00/29134	International filing date (day/month/year) 20 October 2000 (20.10.2000)	Priority date (day/month/year) 20 October 1999 (20.10.1999)
International Patent Classification (IPC) or national classification and IPC IPC(7): B32B 27/16, 27/18, 27/26, 31/28; C08J 3/28; C08K 3/22; C09J 5/06; C08F 2/44, 2/46, 4/04, 4/26. and US Cl.: 156/272.4; 522/ 006, 024, 029, 071, 074, 077, 083, 099, 148.		
Applicant LOCTITE CORPORATION		
<p>1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of <u>4</u> sheets, including this cover sheet.</p> <p><input type="checkbox"/> This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).</p> <p>These annexes consist of a total of <u>0</u> sheets.</p>		
<p>3. This report contains indications relating to the following items:</p> <p>I <input checked="" type="checkbox"/> Basis of the report</p> <p>II <input type="checkbox"/> Priority</p> <p>III <input type="checkbox"/> Non-establishment of report with regard to novelty, inventive step and industrial applicability</p> <p>IV <input type="checkbox"/> Lack of unity of invention</p> <p>V <input checked="" type="checkbox"/> Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</p> <p>VI <input type="checkbox"/> Certain documents cited</p> <p>VII <input type="checkbox"/> Certain defects in the international application</p> <p>VIII <input checked="" type="checkbox"/> Certain observations on the international application</p>		
Date of submission of the demand <u>May 18, 2001</u>	Date of completion of this report 12 March 2002 (12.03.2002)	
Name and mailing address of the IPEA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703)305-3230	Authorized officer  Susan W. Benman Telephone No. 703 308 0661	

I. Basis of the report**1. With regard to the elements of the international application:***

- ☒ the international application as originally filed.
- ☒ the description:
pages 1-26 _____ as originally filed
pages NONE _____, filed with the demand
pages NONE _____, filed with the letter of _____.
- ☒ the claims:
pages 27-30 _____, as originally filed
pages NONE _____, as amended (together with any statement) under Article 19
pages NONE _____, filed with the demand
pages NONE _____, filed with the letter of _____.
- ☒ the drawings:
pages none _____, as originally filed
pages NONE _____, filed with the demand
pages NONE _____, filed with the letter of _____.
- ☐ the sequence listing part of the description:
pages NONE _____, as originally filed
pages NONE _____, filed with the demand
pages NONE _____, filed with the letter of _____.

2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language _____ which is:

- ☐ the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in printed form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. ☒ The amendments have resulted in the cancellation of:

- ☒ the description, pages none
- ☒ the claims, Nos. none
- ☒ the drawings, sheets/fig none

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).**

* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).

** Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.

V. Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**1. STATEMENT**

Novelty (N)	Claims <u>10-19</u>	YES
	Claims <u>1-9,20-30</u>	NO
Inventive Step (IS)	Claims <u>NONE</u>	YES
	Claims <u>1-30</u>	NO
Industrial Applicability (IA)	Claims <u>1-30</u>	YES
	Claims <u>NONE</u>	NO

2. CITATIONS AND EXPLANATIONS

Claims 1-9 and 20-30 lack novelty under PCT Article 33(2) as being anticipated by CLARK, JR. ET AL. CLARK, JR. ET AL. disclose curable compositions comprising between 0.1 and 10 percent by weight microwave absorbable particles in the resulting polymeric composite. The particles, such as ferrite, are selected so that the Curie temperature of the particles is higher than the curing temperature of the curable resin. Thermosetting polymers, including silicones, can be hardened by the method taught, that includes directing microwave energy to a selected portion of the polymeric composite. The claim recitation "about 10 % by weight of said composition" is considered to include 10% by weight of the resulting polymeric composite disclosed by CLARK, JR. ET AL.

Claims 1-17 and 20-30 lack an inventive step under PCT Article 33(3) as being obvious over CLARK, JR. ET AL. in view of TAKAHASHI ET AL.. CLARK, JR. ET AL. teach microwave curable compositions comprising thermosetting polymers and between 0.1 and 10 % by weight ferromagnetic particles. TAKAHASHI ET AL teach adding dielectric materials, such as ferrites, in amounts ranging from 5 to 100 parts by weight, to an organopolysiloxane. TAKAHASHI ET AL teach addition curing an organopolysiloxane composition comprising finely divided silica filler, a peroxide crosslinking agent and a platinum hydrosilation catalyst with microwave energy to provide a cured and foamed silicone rubber. It would not have involved an inventive step to employ the organosiloxane compositions taught by TAKAHASHI ET AL as the thermosetting silicones suggested to be employed in the compositions disclosed by CLARK, JR. ET AL because TAKAHASHI ET AL also teach microwave curing. It would not have involved an inventive step to provide a cured and foamed silicone rubber using particles selected by the Curie temperatures, as taught by CLARK, JR. ET AL, and by adding an amount of 10% or greater dielectric material to ensure sufficient absorption of microwave energy, as taught by TAKAHASHI ET AL. in analogous art.

Claims 1-10 and 13-30 lack an inventive step under PCT Article 33(3) as being obvious over CLARK, JR. ET AL. in view of KITTLE ET AL.. CLARK, JR. ET AL. teach microwave curable compositions comprising thermosetting polymers and between 0.1 and 10% by weight ferromagnetic particles. KITTLE ET AL teach adding from 0.25 to 30 parts by weight microwave sensible material, such as carbon black or metal oxides, to polydiorganosiloxane compositions to be cured by exposure to microwave energy. KITTLE ET AL teach also adding an acetylenic alcohol inhibitor to the polydiorganosiloxane compositions. It would not have involved an inventive step to employ the polydiorganosiloxane compositions taught by KITTLE ET AL as the thermosetting silicones suggested to be employed in the compositions disclosed by CLARK, JR. ET AL because KITTLE ET AL also teach microwave curing. It would not have involved an inventive step to provide a cured and foamed silicone rubber using particles selected by the Curie temperature, as taught by CLARK, JR. ET AL., and by adding an amount of 0.25 to 30% by weight microwave sensible material, as taught by KITTLE ET AL. in analogous art.

Claims 10-19 meet the criteria for novelty set out in PCT Article 33(2). The cited prior art to CLARK, JR. ET AL. does not disclose the combination of heat curable resin, microwave absorbable particles and (1) a curing agent that is an addition curing catalyst or (2) the additives or viscosity modifying agent or inhibitor set forth in claims 10-19.

Claims 1-30 meet the criteria set out in PCT Article 33(4) because the compositions and method are useful for providing and curing polymeric composites.

----- NEW CITATIONS -----

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the questions whether the claims are fully supported by the description, are made:

Claims 1-3 are objected to under PCT Rule 66.2(a)(v) as lacking clarity under PCT Article 6 because the claims are indefinite for the following reason(s): Claim 1 recites microwave absorbable particles in "an amount of about 10% by weight of said composition". Claim 2 recites "an amount sufficient to uniformly cure said compositions". Claim 3 recites "at least about 10% to about 40% by weight of said composition". It is, therefore, not clear what amount of microwave absorbable particles is intended to be present in the composition.